



Control of Interfacial Phenomena in Oxide Heterostructures

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14. ABSTRACT Research has focused on discovering new oxide materials systems with novel properties and demonstrating their potential for high performance electronic applications. We have successfully developed materials with novel interfacial phenomena for defense applications in oxides and their heterostructures. Moreover, we have extended our research to the interfaces of complex oxides and transition metal chalcogenides with novel emergent electronic phases. Our achievements include the discovery of a new conducting channel between two oxides, KTaO3 and LaTiO3, with the highest room temperature electron mobility reported so far for oxide interfaces. We have also explored how to control the properties of complex oxides and their heterostructures using the field effect to control superconductivity, magnetism, and metal-insulator transitions. We also identify the existence of double TiO2 layers at the surface of SrTiO3 in the recently discovered monolayer high temperature superconductor FeSe/SrTiO3. Theoretical studies show that the double TiO2 layers play a crucial role in determining the superconducting states of monolayer FeSe/SrTiO3.						
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Control of interfacial phenomena in artificial oxide heterostructures

Charles H. Ahn

Yale University

Abstract:

For the Air Force, defense applications require electronic devices for sensing, computing, communications, and energy storage based on new materials that provide unique capabilities or superior performance. Novel phases that arise at interfaces between complex oxide materials provide a promising pathway to realizing new classes of devices that exhibit functionalities not found in conventional technologies. New materials based on perovskite oxides are promising because of their wide variety of properties, including electronic conduction, superconductivity, magnetic response, and optical properties. Research has focused on discovering new oxide materials systems with novel properties and demonstrating their potential for high performance electronic applications. We have successfully developed materials with novel interfacial phenomena for defense applications in oxides and their heterostructures. Moreover, we have extended our research to the interfaces of complex oxides and transition metal chalcogenides with novel emergent electronic phases. Our achievements include the discovery of a new conducting channel between two oxides, KTaO_3 and LaTiO_3 , with the highest room temperature electron mobility reported so far for oxide interfaces. We have also explored how to control of the properties of complex oxides and their heterostructures using the field effect to control superconductivity, magnetism, and metal-insulator transitions. Such control is critical for the development of oxide-based electronic devices. We also identify the existence of double TiO_2 layers at the surface of SrTiO_3 in the recently discovered monolayer high temperature superconductor FeSe/SrTiO_3 . Theoretical studies show that the double TiO_2 layers play a crucial role in determining the superconducting states of monolayer FeSe/SrTiO_3 . Our research in these projects has developed new materials systems with unique properties and has led to progress in achieving high performance electronic applications based on complex oxides.

Our approach to elucidate and manipulate new materials phases at complex oxide interfaces focuses on growth and characterization using state-of-the-art techniques. The growth of complex oxides is performed using molecular beam epitaxy (MBE) facilities within the research group of the PI. Various characterization techniques are performed at Yale and at user facilities located at Brookhaven and Argonne National Laboratories.

We have pursued multiple projects funded by AFOSR. The first project resulted in the discovery of a new conducting 2-dimensional electron system between two complex oxides, KTaO_3 and LaTiO_3 , both of which are insulating in bulk. This conducting channel has the highest room

temperature electron mobility reported so far for oxide interfaces. Another project explores general approaches to control the properties of oxides and their heterostructures by the field effect. Applying a gate voltage through gate insulators leads to a modulation of the carrier density and properties sensitive to carrier density, such as magnetism and metal-insulator transitions. Approaches to control these properties are essential for the development of complex oxide-based electronic devices. A third project is an experimental and theoretical study of a recently discovered high temperature superconductor, monolayer FeSe/SrTiO₃ (STO). For the first time, we identify the existence of double TiO₂ layers at the surface of STO. The double TiO₂ layers play a crucial role in determining the superconducting states of monolayer FeSe/STO.

Below, we describe the three projects in detail:

1. Discovery of a new conducting channel at LaTiO₃/KTaO₃ interfaces.

The dominant operation mode of current electronics devices relies on the control of conduction channels in conventional semiconductors, such as Si. The electronic properties of these channels, including electron carrier density and mobility, determine the performance of the devices. One promising and versatile approach to achieving high carrier densities is to use interfaces involving perovskite oxide ABO₃ heterostructures. So far, only SrTiO₃ (STO) has been engineered to serve as the host for high density 2-dimensional electron gases (2DEGs), such as in LaAlO₃ (LAO)/STO. The sheet carrier density in LAO/STO can be as high as $2 \times 10^{13} \text{ cm}^{-2}$, which is difficult to achieve in conventional semiconductors. The carrier mobility in LAO/STO also reaches 800-10,000 cm²/Vs at low temperatures. However, one serious technological limitation of STO-based conducting oxide interfaces for electronics applications is the relatively low carrier mobility (0.5 - 10 cm²/Vs) of STO at room temperature.

Motivated by the need for higher mobility systems, we investigate an alternate host for high carrier density 2DEGs, KTaO₃ (KTO). Scanning transmission electron microscopy images of the interfaces (Fig. 1(a)) show that the LaTiO₃ (LTO) films are epitaxial on the KTO substrates and form a fully crystalline interface. For LTO/KTO interfaces, we observe metallic conduction from T = 2 K to room temperature (300 K) and electron densities in the order of $1 \times 10^{14} \text{ cm}^{-2}$ (Fig. 1(b)). The electron densities achieved are comparable to the densities in STO based heterostructures and are higher than in conventional semiconductors.

By using KTO, we achieve mobilities in LTO/KTO interfaces as high as 21 cm²/Vs at room temperature, over a factor of 3 higher than observed in doped bulk STO (Fig. 1(c)). In the Drude model, the carrier mobility μ depends on the scattering time τ and effective mass m^* as $\mu = e\tau/m^*$, where e is the electronic charge. The conduction bands in STO heterostructures have primarily Ti 3d character, while the conduction bands in KTO have primarily Ta 5d character. *Ab initio* calculations confirm the formation of a 2DEG in the interfacial LTO/KTO

layer that resides in bands having Ta 5d character. We calculate an electron effective mass m^*/m_e of 0.34 for the lowest energy interfacial Ta 5d-dominated bands, which should be compared to 0.49 for 2DEGs in STO. We attribute the higher mobility in the KTO 2DEGs, compared to STO 2DEGs, to the smaller effective mass for electrons in KTO in these bands.

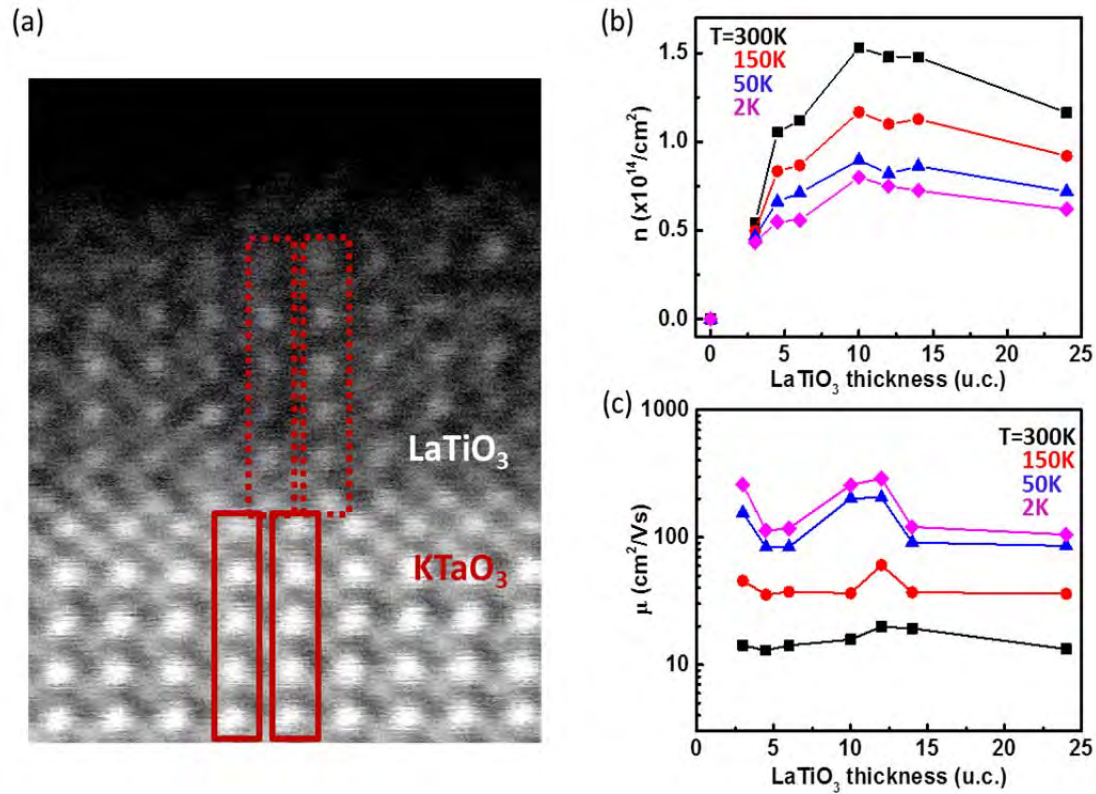


Figure 1. (a) Scanning transmission electron microscopy image showing the crystal structure of a 6uc LaTiO₃/KTaO₃ heterostructure. The red boxes are a guide to the eye and highlight the heavier atoms (La (dashed line) and Ta (solid line)). From the relative locations of Ta and La at the interface, we find that the interface is TaO₂ terminated. (b), (c) Thickness dependence of electron density n and mobility μ of LaTiO₃/KTaO₃ at 300 K (black), 150 K (red), 50 K (blue) and 2 K (magenta). The lines connecting the data are a guide to the eye.

Reference: K. Zou, S. Ismail-Beigi, K. Kisslinger, X. Shen, D. Su, F.J. Walker, and C.H. Ahn, LaTiO₃/KTaO₃ interfaces: A new two-dimensional electron gas system. *APL Materials*, 3, 036104, (2015). DOI: 10.1063/1.4914310.

2. Controlling oxide based devices by field effect.

Successful identification of interfaces exhibiting tunable properties is the first step to integrate oxide materials into devices. A subsequent step is to control the properties and optimize the performance of the oxide-based electronic devices. The performance of the complementary metal oxide semiconductor (CMOS) transistors ubiquitously found in electronic devices has reached a plateau. This plateau in performance is due in part to the fundamental materials constraints of Si-based technology. Limitations in carrier mobility and fluctuations in carrier density can contribute to short-channel effects, which become more pronounced as transistors are scaled to smaller dimensions. Complex oxides are being developed for post-CMOS electronics. Key progress in materials growth and device fabrication has opened new pathways to control correlated phenomena through applied electric fields (Fig. 2). Field-effect transistors in which magnetism, superconductivity, and metal-insulator transitions can be controlled have become a reality.

We describe these key developments and outline future directions to be taken in the development of complex oxide devices. Emphasis is given to our work on tuning the properties of both the bulk oxides and the heterostructures.

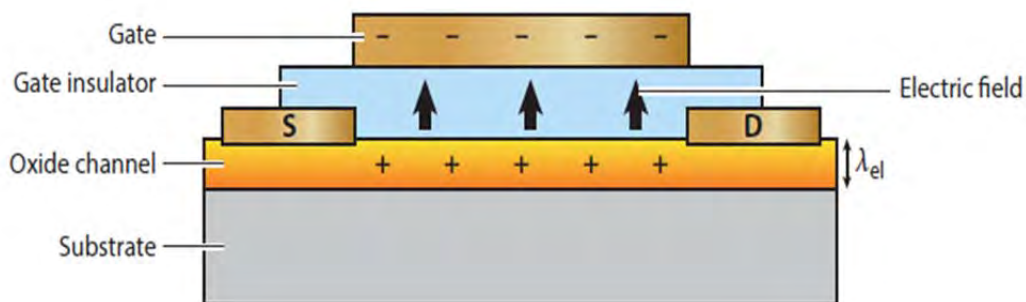


Figure 2. Diagram of electric-field-effect devices. By applying an electric field across the gate insulator, we can effectively modulate the carrier density in the oxide channel and further control other properties.

Reference: J. Ngai, F.J. Walker, and C.H. Ahn, Correlated Oxide Physics and Electronics. *Annual Review of Materials Research*, 44, 1, (2014). DOI: 10.1146/annurev-matsci-070813-113248.

3. Structural studies of a high temperature superconductor, monolayer FeSe/SrTiO₃.

Superconductors that operate at elevated temperatures without loss or heat generation have a range of applications, from nanoscale devices to macroscale power transmission. Finding new high temperature superconductors and understanding their origin are current topics in condensed matter physics with clear applications to electronic devices.

The discovery of the iron-based superconductors exhibiting unconventional superconductivity promises to enhance our understanding and lead to the development of new materials. A unique superconducting state exists at the interface of a monolayer of FeSe grown on SrTiO₃ (STO) with a critical temperature, T_c , up to 109 K. Because only a single monolayer is superconducting, while multiple layers are not, we conclude that interactions at the interface play an important role in the existence of superconductivity. However, a complete understanding of the relationship between the superconducting state and the structure of monolayer FeSe has not been elucidated. The goal of this project is to experimentally determine the physical structure at the interface and the role of charge carrier doping for the superconducting phase.

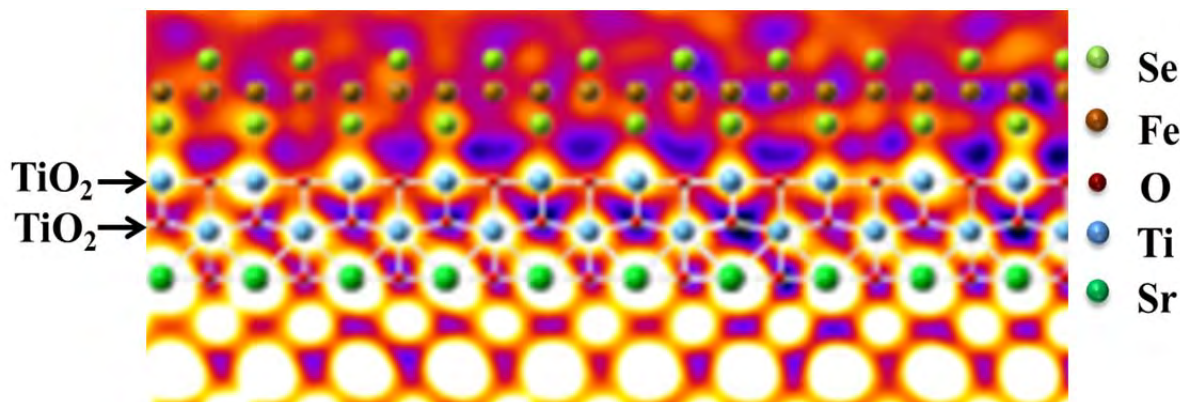


Figure 3. Scanning transmission electron microscopy image showing the crystal structure of an FeSe/SrTiO₃ interface with double TiO₂ layers. The symbols of atoms are a guide to the eye.

Monolayer FeSe is grown in a molecular beam epitaxy system dedicated to the growth of chalcogenides. The substrates are prepared using a high temperature anneal in oxygen at ambient pressure. This procedure typically results in a double TiO₂ termination. We determine the interfacial structure using scanning transmission electron microscopy (Fig. 3) and synchrotron x-ray diffraction at Brookhaven and Argonne National Laboratories (Fig. 4(a)). We identify the surface reconstruction of STO present at a monolayer FeSe/STO interface as $\sqrt{13} \times \sqrt{13}$ R33.7 (Fig. 4(a)). This reconstruction is not just a rearrangement of the surface

atoms of a bulk truncation, but is a change in surface stoichiometry, where the surface has a double TiO_2 surface termination (Fig. 3).

We show that this reconstruction is critical in two significant ways. First, this reconstruction facilitates the growth of a coherently strained, epitaxial FeSe monolayer, while a bulk terminated surface does not. Growth on surfaces terminated with a single layer of TiO_2 results in highly disordered films. Monolayer FeSe samples grown on the reconstructed surfaces are epitaxial and coherently strained, as observed using reflection high-energy electron diffraction and synchrotron x-ray diffraction.

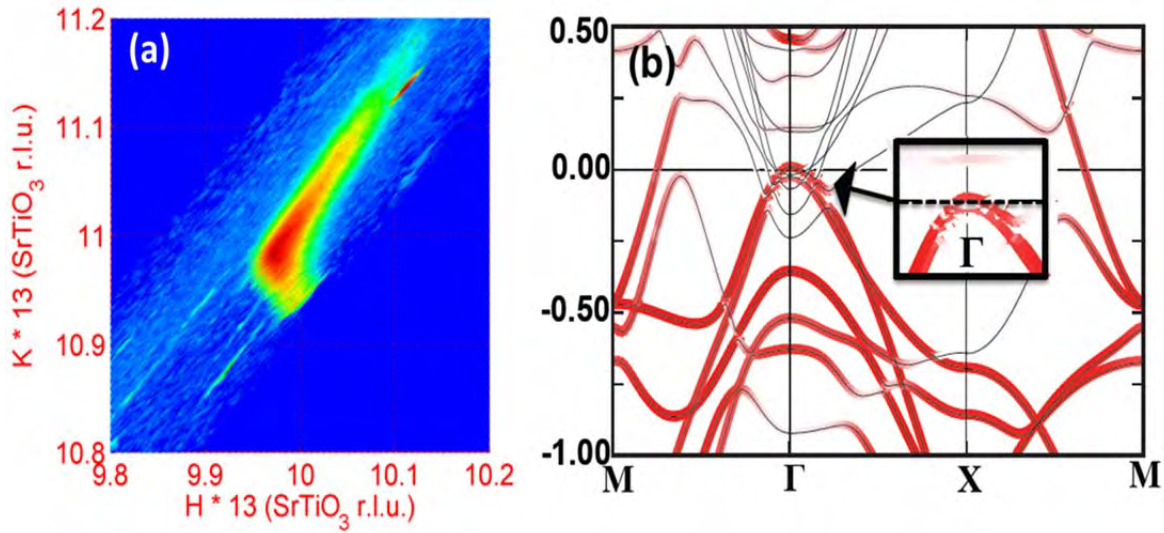


Figure 4. (a) Reciprocal space maps showing $[10/13, 11/13, 0.8]$ SrTiO_3 superstructure reflections for 1 monolayer FeSe/ SrTiO_3 capped with 10nm Se. (b) Orbitally resolved band structures for Fe-3d for four variants of an FeSe monolayer on double TiO_2 terminated SrTiO_3 with 50% oxygen vacancies. Thin black curves represent bands for the whole system, while the red curves are projections of the Fe-3d orbital. The Fermi level is set to zero in each case. The energy scale is in eV. The inset represents the zoom-in view of Fe bands around the Γ -point in a small energy window.

Second, in collaboration with S. Ismail-Beigi at Yale, we calculated the band structure of FeSe on single and double TiO_2 terminated STO, in order to explore the role of double TiO_2 surface structures on the electronic structure of FeSe/STO. These calculations suggest that the reconstructed surface facilitates the formation of oxygen vacancies and electron transfer to the FeSe monolayer by modifying both the defect chemistry and electronic structure at the FeSe-STO interface. One unique feature of the experimentally determined electronic band structure of superconducting monolayer FeSe/STO is that the hole pocket at the Γ point is not present, as it is for bulk or thick FeSe films. We compute band structure for monolayer FeSe/STO with a

fully stoichiometric interface and find the hole pocket at the Γ point, in agreement with angle-resolved photoemission spectroscopy (ARPES) measurements of thick films. Oxygen vacancies dope the FeSe with electrons and move the Fermi level upwards (Fig. 4 (b)). With a double TiO_2 layer termination, this doping opens a gap at the Γ point and removes the hole pocket, recovering the band structure observed in ARPES (Fig. 4 (b)).

The importance of a reconstructed surface has not been previously considered in theories of monolayer FeSe superconductivity and appears to be present in previous reports of monolayer superconductivity on the STO surface before growth. This work provides compelling evidence that the charge transfer facilitated by the double TiO_2 layers is critical for the superconductivity in monolayer FeSe/STO. There are still many open questions about this novel superconductor that likely rely on a detailed knowledge of the interface structure determined here.

Reference: K. Zou, S. Mandal, F.J. Walker, Sohrab Ismail-Beigi, C.H. Ahn, et al. The crucial role of double TiO_2 layers at the interfaces of FeSe/ SrTiO_3 superconductors, *in preparation*.

Presentations:

“ $\text{LaTiO}_3/\text{KTaO}_3$ interfaces: A new two-dimensional electron gas system”, International Workshop on Recent Progress in the Functionality of Artificial Oxide Structures, Institute of Physics, CAS, Beijing, China. (Invited Talk)

“Extreme carrier concentrations and metallic conduction in thin LaTiO_3 films”, 21st International Workshop on Oxide Electronics, Bolton Landing, NY. (Poster)

“High room temperature carrier density and mobility in $\text{LaTiO}_3/\text{KTaO}_3$ heterostructures”, Materials Research Society (MRS) Spring Meeting 2014, San Francisco, CA. (Contributed Talk)

“Growth and transport studies of $\text{LaTiO}_3/\text{KTaO}_3$ heterostructures”, American Physical Society (APS) March Meeting, Denver, CO. (Contributed Talk)

“Extreme carrier concentrations and metallic conduction in thin LaTiO_3 films”, Materials Research Society (MRS) Fall Meeting 2013, Boston, MA. (Contributed Talk)

Publications:

K. Zou, S. Ismail-Beigi, K. Kisslinger, X. Shen, D. Su, F.J. Walker, and C.H. Ahn, $\text{LaTiO}_3/\text{KTaO}_3$ interfaces: A new two-dimensional electron gas system. *APL Materials*, 3, 036104, (2015). DOI: 10.1063/1.4914310.

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K. Zou, S. Mandal, F.J. Walker, Sohrab Ismail-Beigi, C.H. Ahn, et al., The crucial role of double TiO_2 layers at the interfaces of FeSe/ SrTiO_3 superconductors, *in preparation*.

1.

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Abstract

For the Air Force, defense applications require electronic devices for sensing, computing, communications, and energy storage based on new materials that provide unique capabilities or superior performance. Novel phases that arise at interfaces between complex oxide materials provide a promising pathway to realizing new classes of devices that exhibit functionalities not found in conventional technologies. New materials based on perovskite oxides are promising because of their wide variety of properties, including electronic conduction, superconductivity, magnetic response, and optical properties. Research has focused on discovering new oxide materials systems with novel properties and demonstrating their potential for high performance electronic applications. We have successfully developed materials with novel interfacial phenomena for defense applications in oxides and their heterostructures. Moreover, we have extended our research to the interfaces of complex oxides and transition metal chalcogenides with novel emergent electronic phases. Our achievements include the discovery of a new conducting channel between two oxides, KTaO₃ and LaTiO₃, with the highest room temperature electron mobility reported so far for oxide interfaces. We have also explored how to control of the properties of complex oxides and their heterostructures using the field effect to control superconductivity, magnetism, and metal-insulator transitions. Such control is critical for the development of oxide-based electronic devices. We also identify the existence of double TiO₂ layers at the surface of SrTiO₃ in the recently discovered monolayer high

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